Characterization of SiC/SiNC CMC Specimens Containing Embedded SiC Disks

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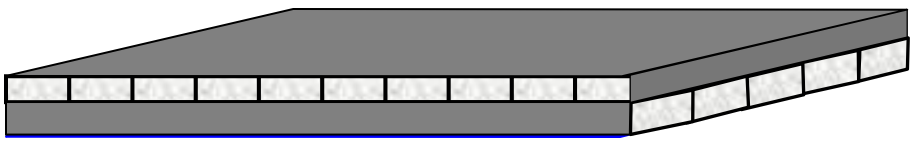

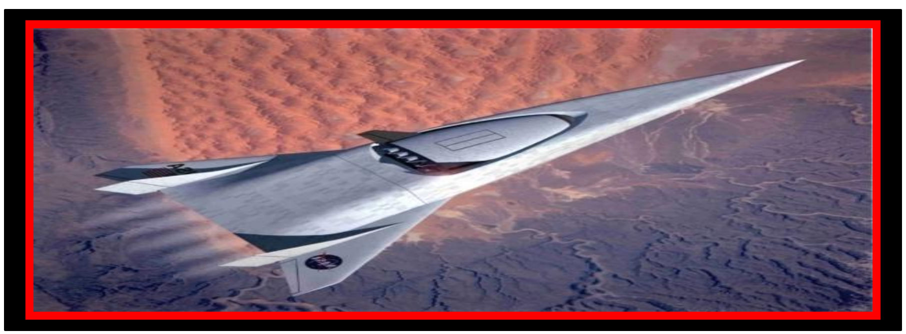

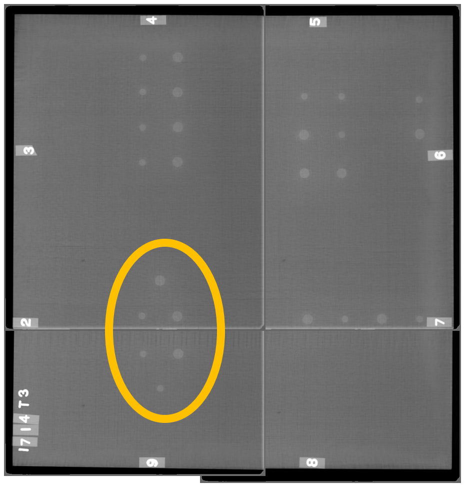

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Hypersonic Materials and Structures (M&S)

Outline

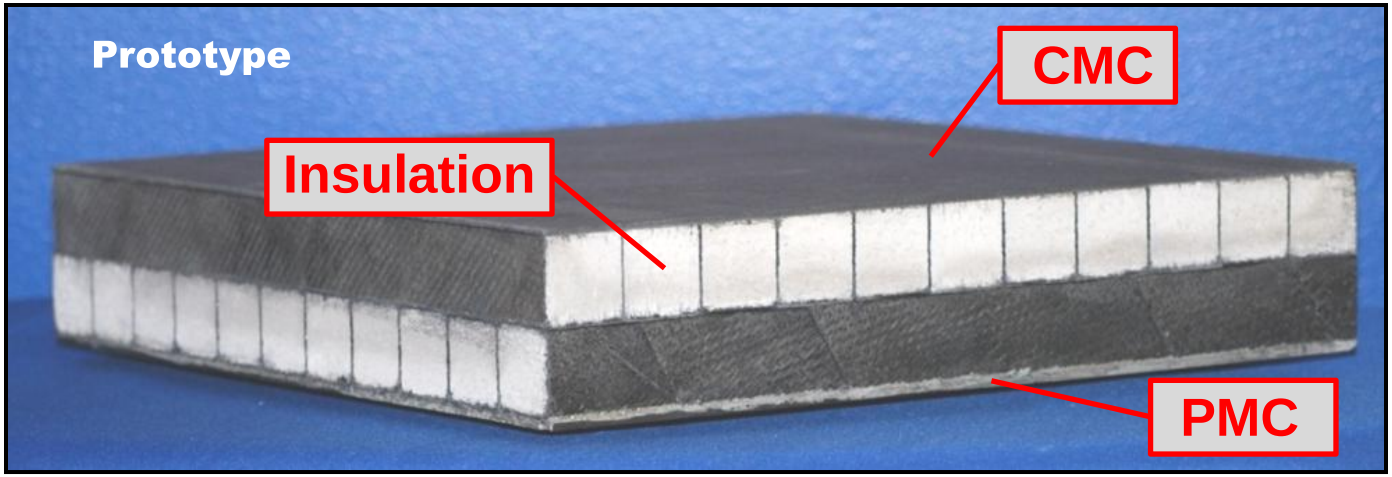
- SITPS and the Embedded Sensor Task
- Creating the “Sensor Panel”
- Characterizing the “Sensor Panel”
- Test specimens—machining and NDE
- Room temperature tensile tests
- Tensile creep tests
- Interlaminar tensile tests
- Summary and Conclusions

Structurally Integrated Thermal Protection Systems (SITPS)

A NASA-led interdisciplinary team with members from several NASA centers and Industry has been developing SITPS (structurally integrated thermal protection system) for use on hypersonic vehicles.

Structurally Integrated Thermal Protection Systems (SITPS)

"A TPS that has both an integrated mechanical and thermal load carrying capability and has the ability to share mechanical loads with adjacent TPS structures"



Driver for NASA's SITPS development:
The development of an advanced TPS that is both structurally and volumetrically efficient using high-temperature ceramic matrix composite and light-weight insulation materials

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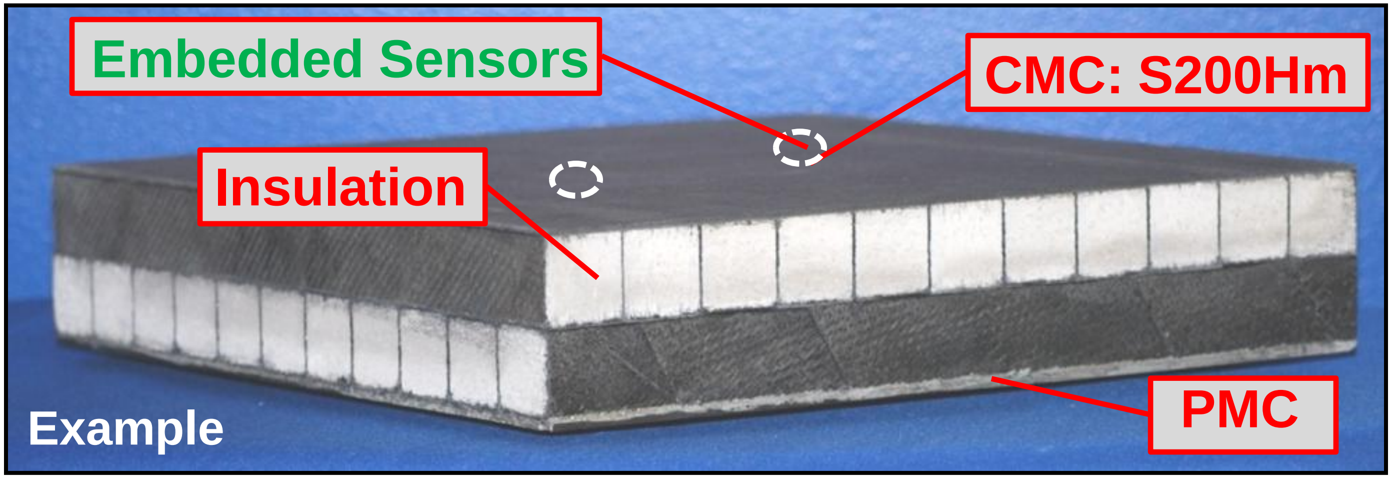
Hypersonics SITPS Embedded Sensors Subtask

Vision

Fully-functional embedded wireless sensors for use in SITPS (structurally integrated thermal protection system)—capable of transmitting and receiving information from within a CMC (ceramic matrix composite) surface layer operating at temperatures above 2000ºF (1093ºC).



Objectives (Note: Two Parallel Efforts Within Task)

→ **There are Materials and Sensors Aspects, With Overlap**

- Characterize embedded SiC "dummy" chips in representative SITPS OML (outer mold line) composite material to help us understand the thermal and mechanical interaction between the SiC chips (including the metallization) and the composite.
- Understanding this interaction is the first step in assessing the feasibility of embedding sensors in SITPS (specifically, in the CMC outer layer).
- Characterize the functional parameters of GRC-developed high-temperature strain gauges in a surface-mounted configuration.
- Develop roadmap for subtask. Chart a path toward surface-mounted wireless sensors followed by embedded wireless sensors.

Surface Mounted Sensors → **Surface Mounted Wireless Sensors** → **Embedded Wireless Sensors**

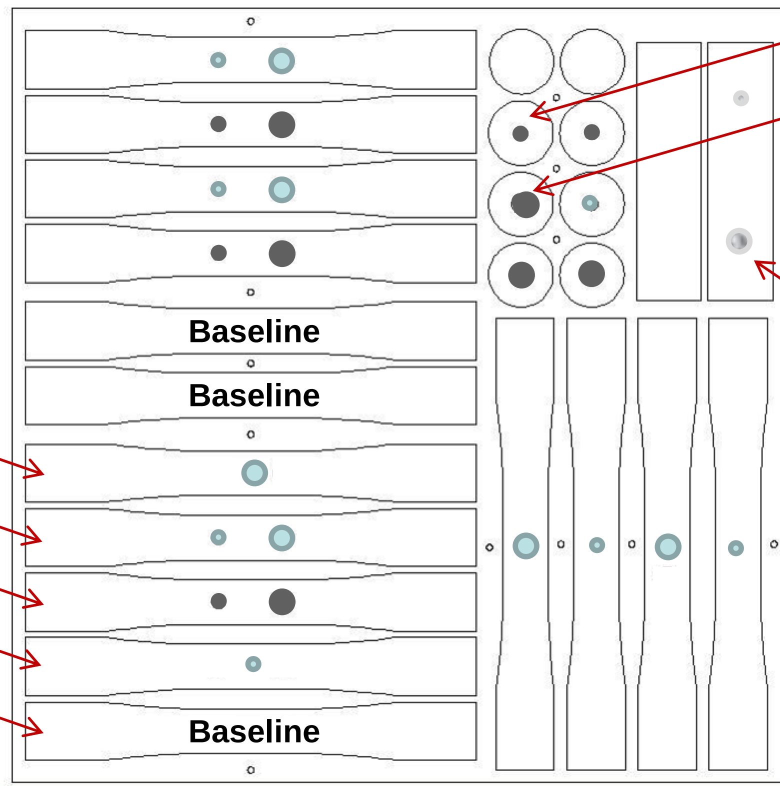
→ **Level of Difficulty (Especially Considering the Operating Temperature)**

Fabrication of the “Sensor Panel”

- ATK COI Ceramics, Inc. fabricated a 12.5 x 12.5” (≈32 x 32 cm) 6-ply SiC/SiNC test panel (S200Hm CMC, similar to the current typical SITPS CMC outer layer) with embedded SiC dummy chips for NASA.

“Dummy Sensors”

- **W-Ni coated**
- **6H SiC**
- **Polycrystalline SiC**



Have 3 of each type of ILT sample

For evaluation of stability of metallic coating

Have 3 of each type of tensile sample

Specimen Layout

Technical Challenges Addressed:

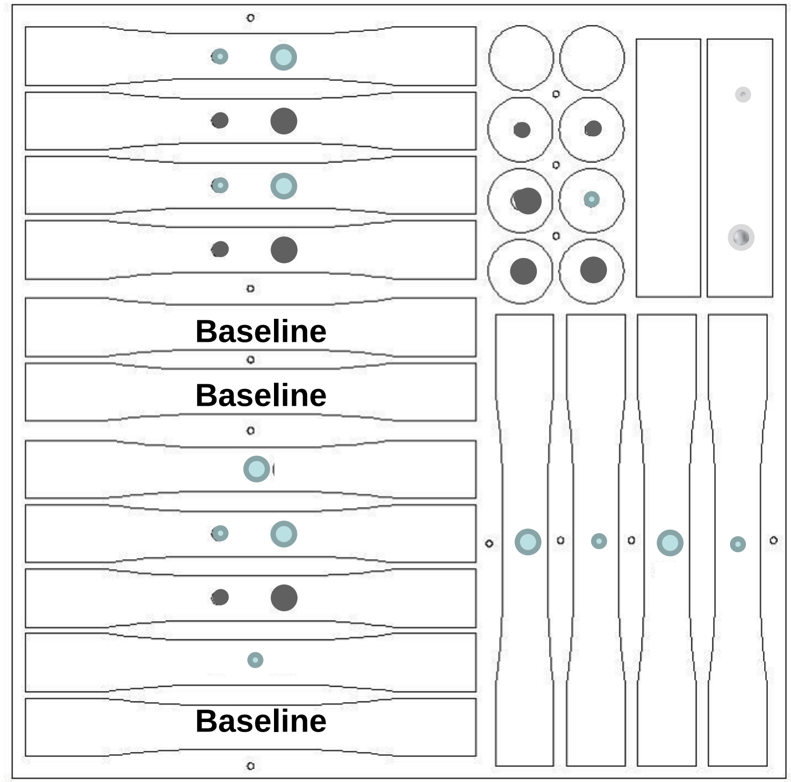
- Fabrication of panel, with accurate placement of test structures and sensors
- Achieving material combinations that can survive 1200°C (2200°F)

Fabrication of the "Sensor Panel"

- Three types of SiC disks (dummy sensors) were provided to COIC by NASA GRC.
- COIC used a template to position the disks.

"Dummy Sensors"

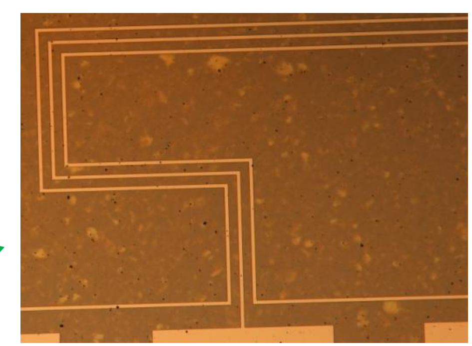
- W-Ni coated
- 6H SiC
- Polycrystalline SiC



- We are interested in seeing if metallic species (W-Ni) deposited on two of the chips will survive the PIP (polymer infiltration/pyrolysis) processing.
- Dimensions of the dummy sensors are as follows:
  Large diameter: 7.5 mm = 0.295 in., t = 10 mils
  Small diameter: 5 mm = 0.197 in., t = 10 mils

Types of SiC disks are as follows:

- **Single crystal 6H SiC:** Purchased in wafer form and machined into chips using a deep reactive ion etcher machine.
- **Polycrystalline SiC:** Purchased in wafer form and machined into chips using a deep reactive ion etcher machine.
- **Chips with patterns on the surface:** Patterns made of a W-Ni metal layer capped with a thinner Si metal layer (~1075 angstroms total thickness). Patterned using a lift-off process.



Panel Characterization Prior to Machining of Specimens

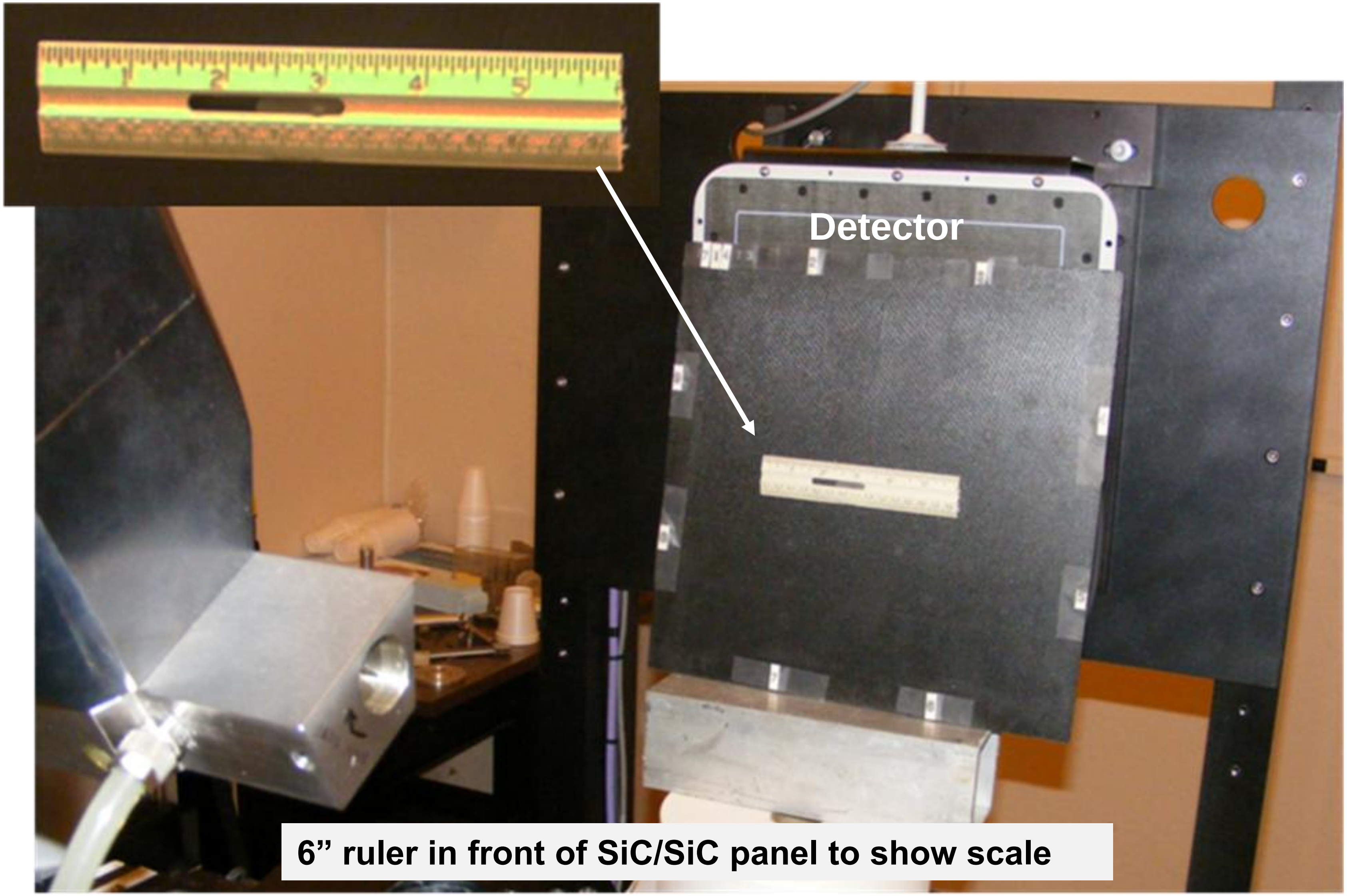
- "Sensor Panel" was evaluated with nondestructive evaluation (NDE) to (A) characterize the embedded chips/surrounding composite material and (B) to determine the location of the dummy sensors (prior to machining samples.

(A) <u>Thermography</u>

- Thermography was selected for the initial characterization (at LaRC) based on factors including the size of panel, potential resolution of differences within the panel, and concerns about immersing the panel in a liquid (avoided w/thermography, which is a "non-contact" approach).
- Of strong interest: the bonding between the S200Hm matrix and the dummy sensors. NDE performed at GRC had yielded definitive information on sensor location, but radiography does not provide information about the bonding.
- While standards do not exist for this system, LaRC NDE characterization provided indications of a difference in the imaging of some of the disks (embedded dummy sensors). CT scans of those disks were performed after the panel was machined.
- Concluded that in subsequent efforts, thermographic evaluation of smaller regions would be better than trying to characterize the entire panel at once.

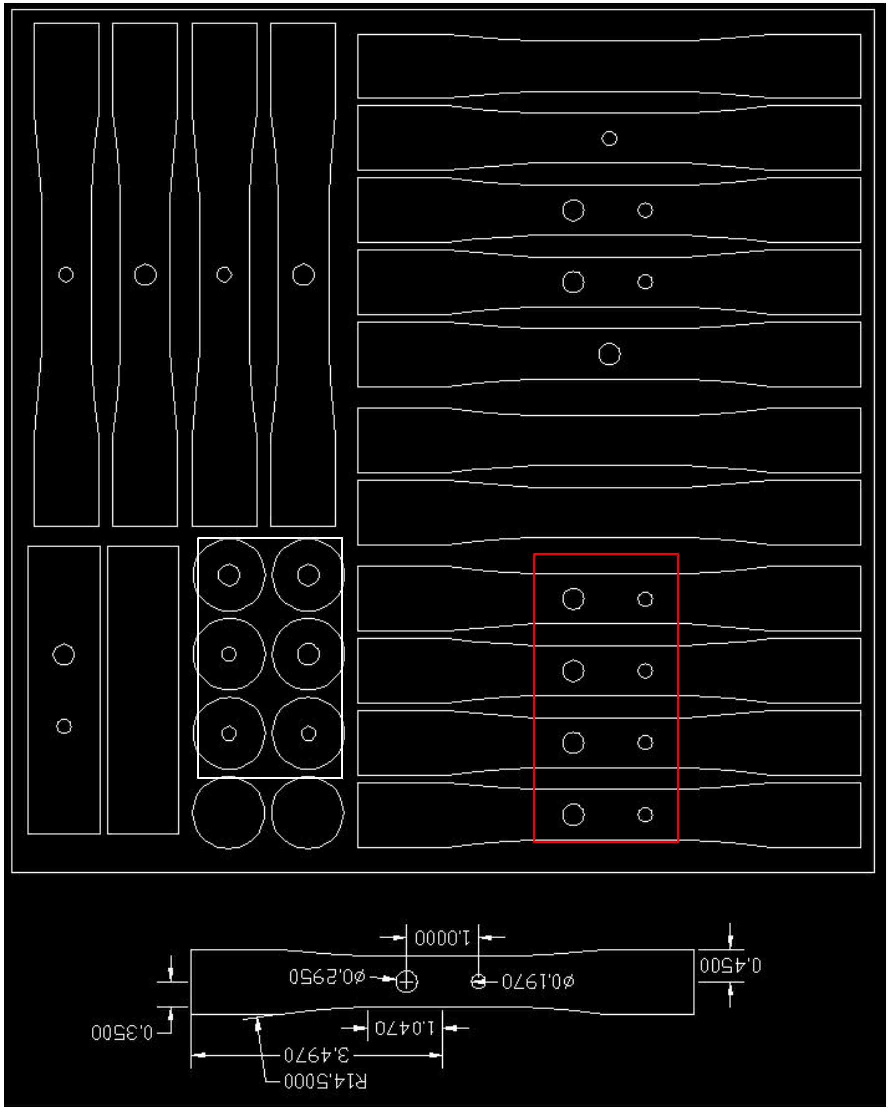
Panel Characterization Prior to Machining of Specimens

Radiographic Test Set-up at NASA GRC

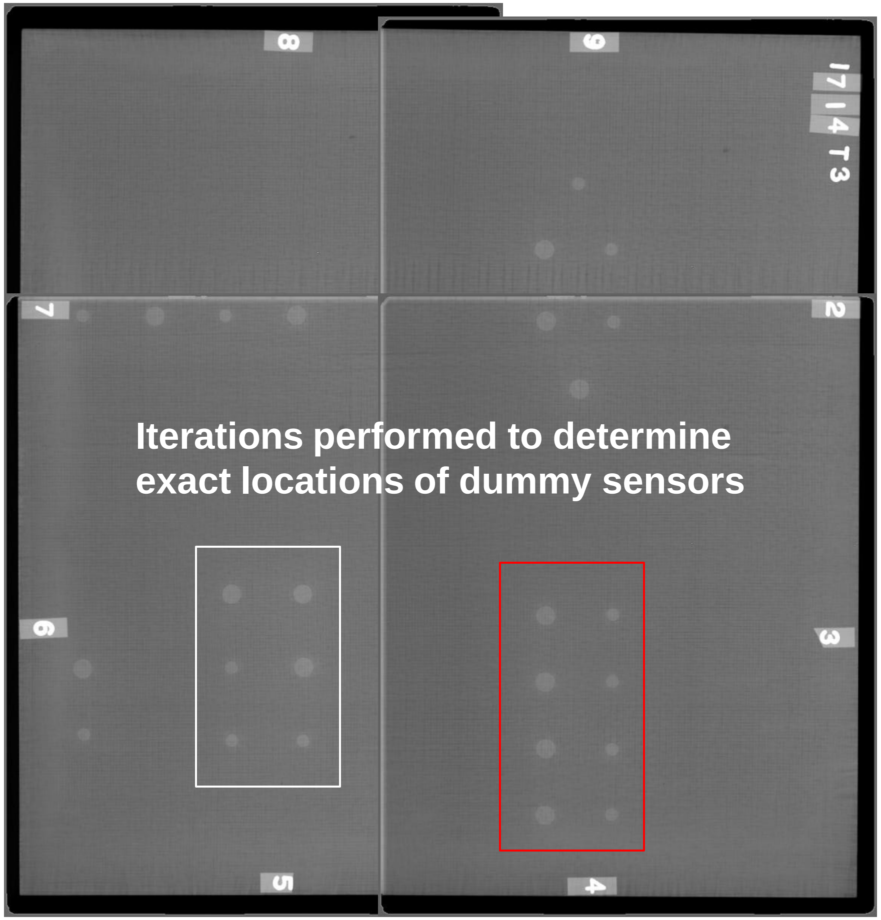


6” ruler in front of SiC/SiC panel to show scale

Panel Characterization Prior to Machining of Specimens



Sensor Panel Layout

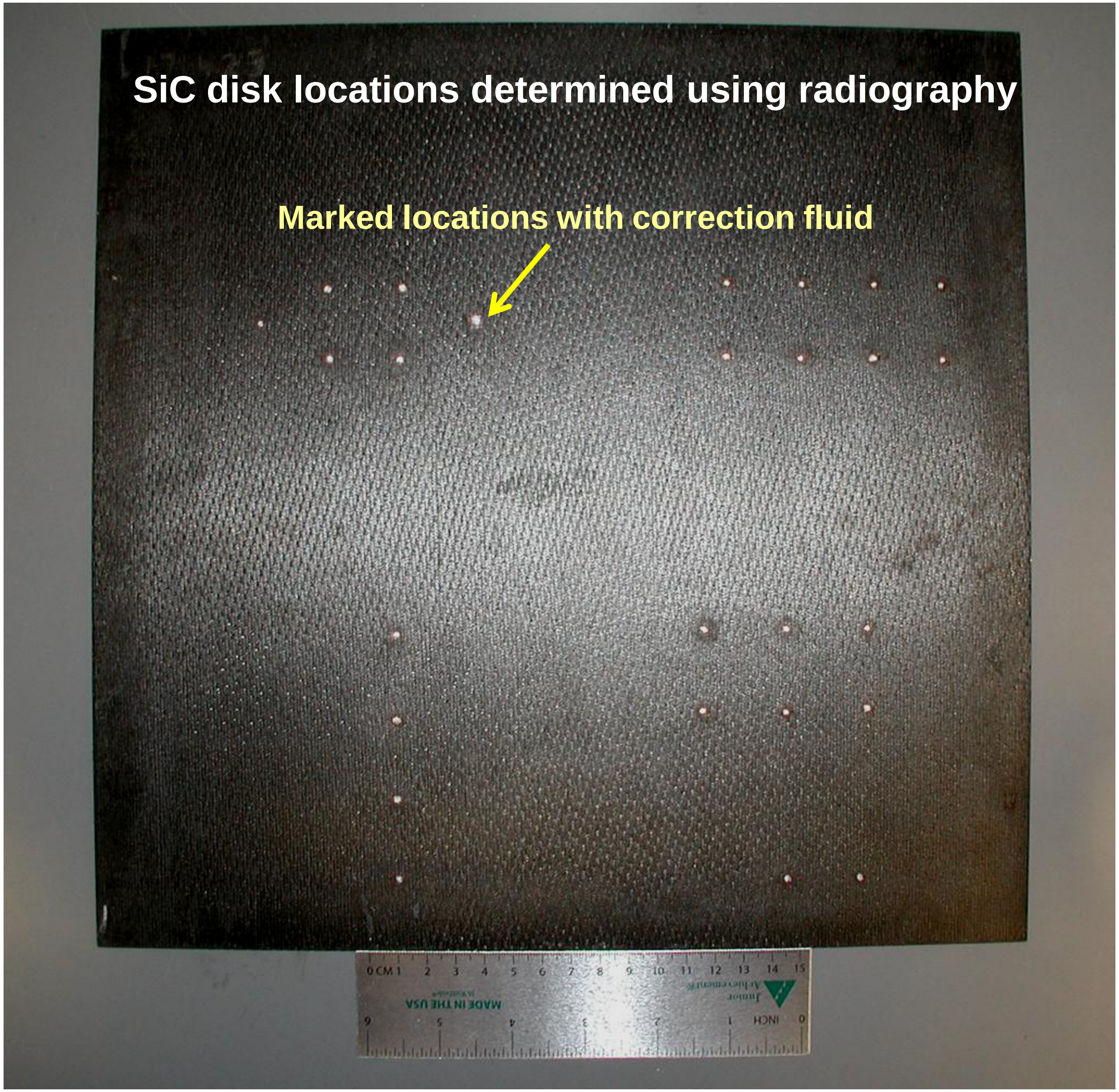


325 mm

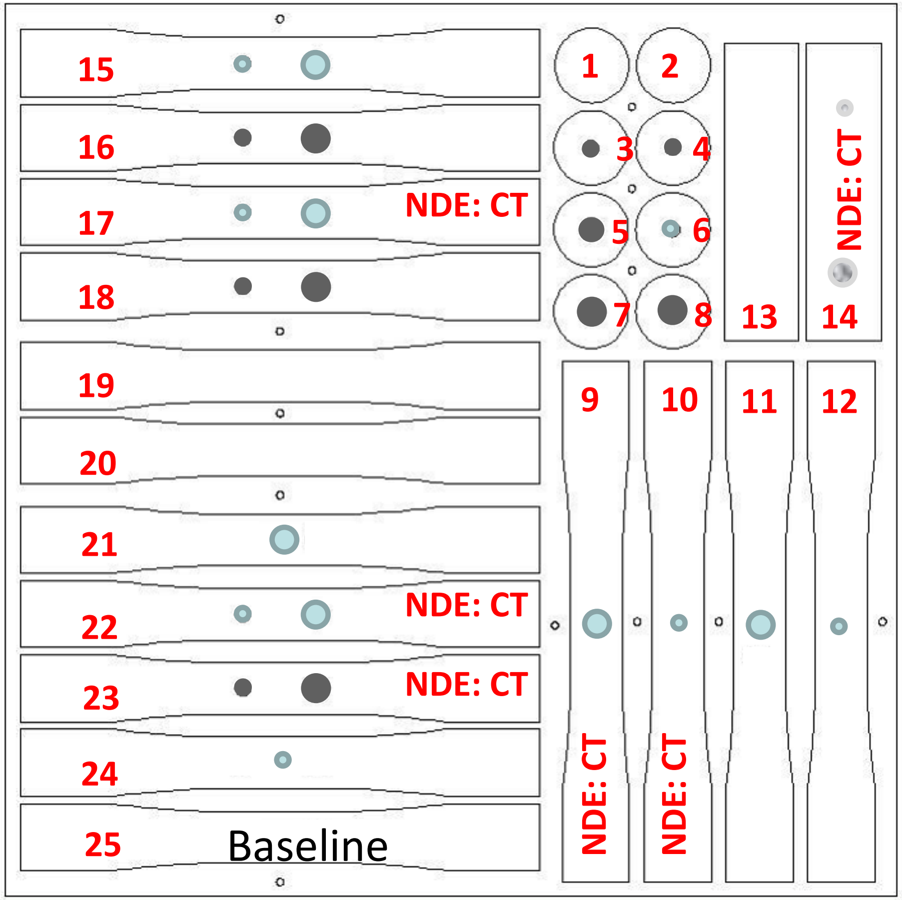
Composite of radiographic results

NDE performed at NASA GRC

Panel Characterization Prior to Machining of Specimens



Machining of Specimens and Further NDE of Specimens



Specimen Layout

- **15 tensile samples were produced (5 variations)**
- **8 ILT (interlaminar tensile) samples were also produced**

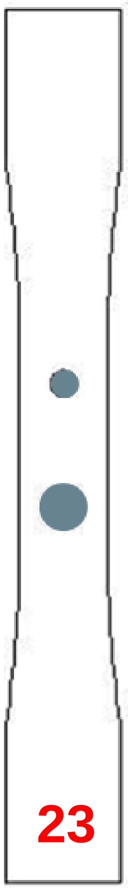
Further NDE of Specimens

NASA LaRC Microfocus X-ray Computed Tomography System

HMXST High Performance Real-Time X-ray Inspection System

- Advanced microfocus x-ray system, capable of resolving details down to 5 microns, with magnifications up to 160X.
- Sample can be manipulated with 5 axes of freedom, while continuously viewing the image on a monitor.
- Defects/features of interest can be rapidly located, zooming in for detailed analysis.
- System is supplied as a complete, large dimension radiation enclosure, with x-ray, manipulator and imaging controls housed in a separate control console.

Computed Tomography of Samples Prior to Testing



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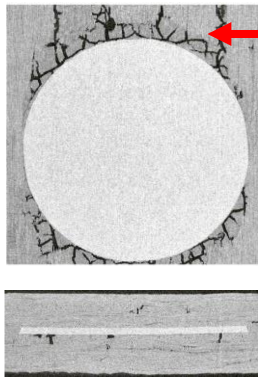
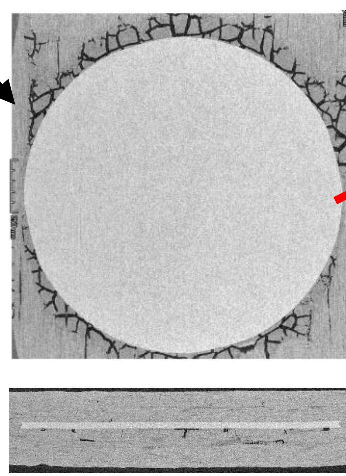


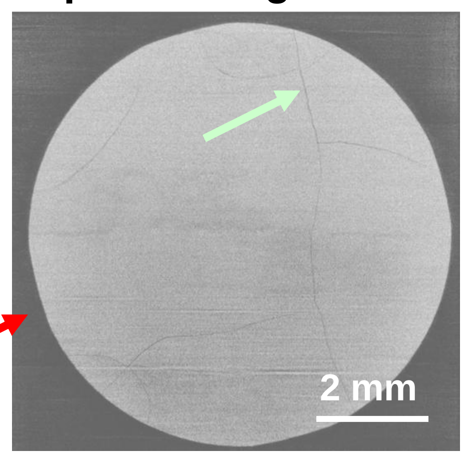
2 mm

The matrix surrounding the disks exhibited cracking



2 mm

Almost all SiC disks were cracked after processing

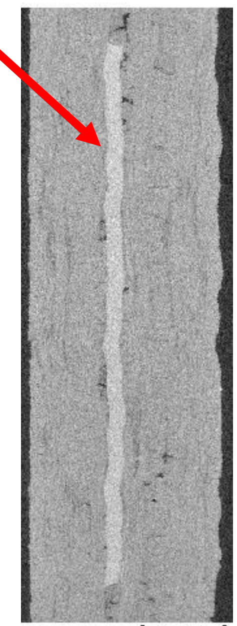


CT slices through the embedded SiC disks

Some samples had large porosity above or below the disk

Some disks appeared deformed and wavy after processing

10

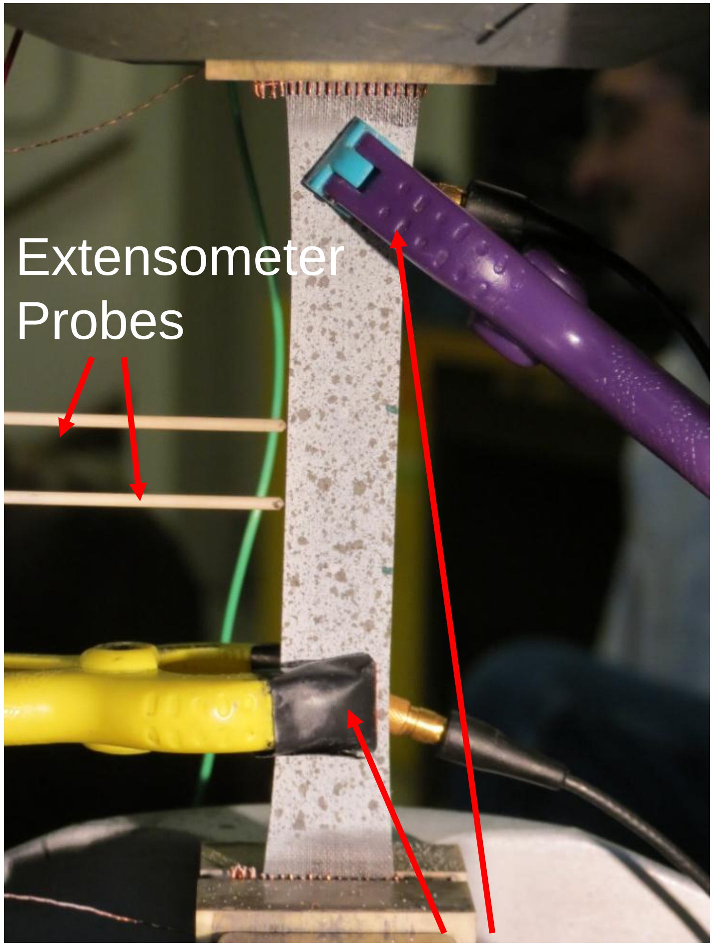


17

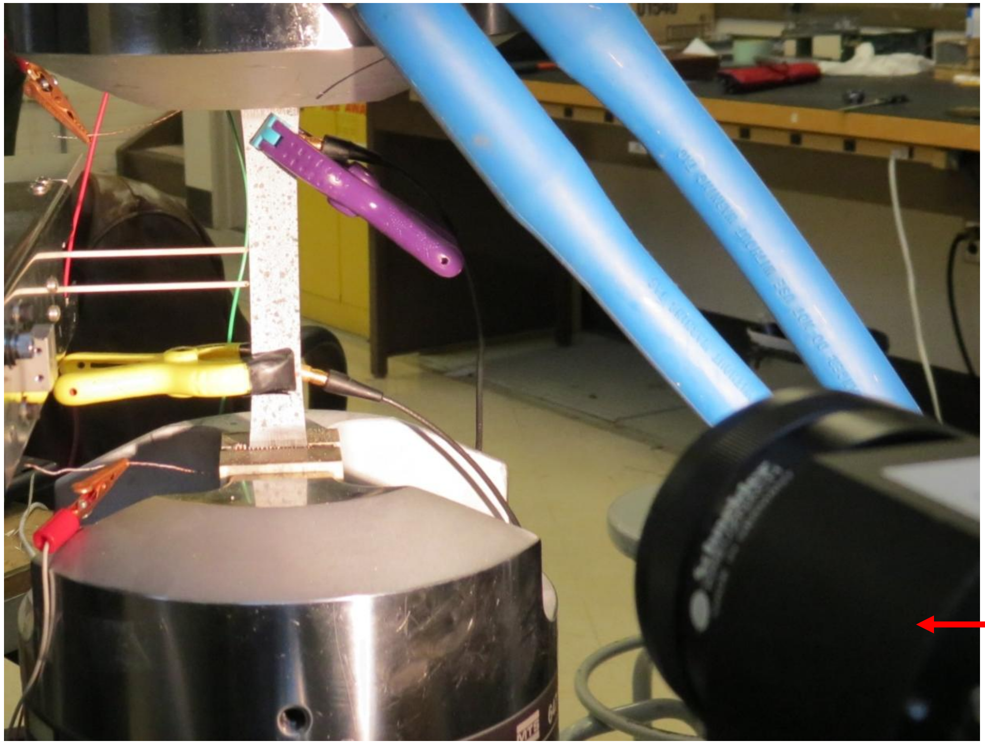
1 mm

Room Temperature Tensile Tests

- Acoustic Emission (AE) monitored throughout test
- Digital Image Correlation (DIC) software utilized for strain visualization
- Contact probe extensometer with 12.7 mm gage and 4% range used
- Tensile specimens loaded at 4 kN/min



Acoustic Emission Sensors

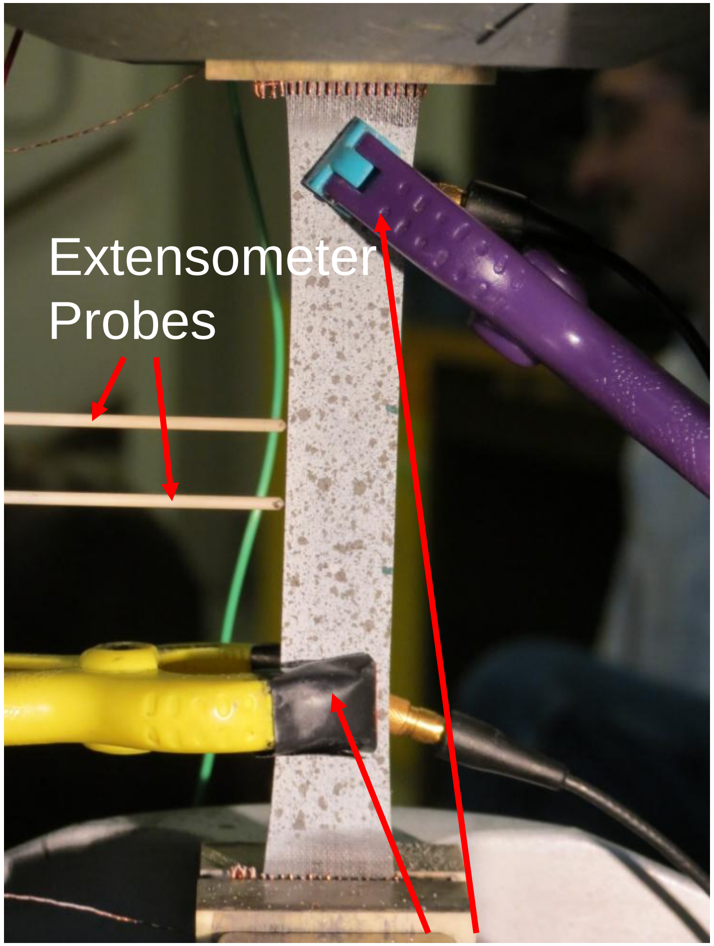


Digital camera:
strain visualization

Room Temperature Tensile Tests

<u>Acoustic Emission</u>

- Method used to monitor matrix cracking in CMCs.
- When cracks form, energy is released and detected by piezoelectric sensors.
- By monitoring the acoustic emission throughout the loading (testing), the crack density as a function of stress can be estimated.

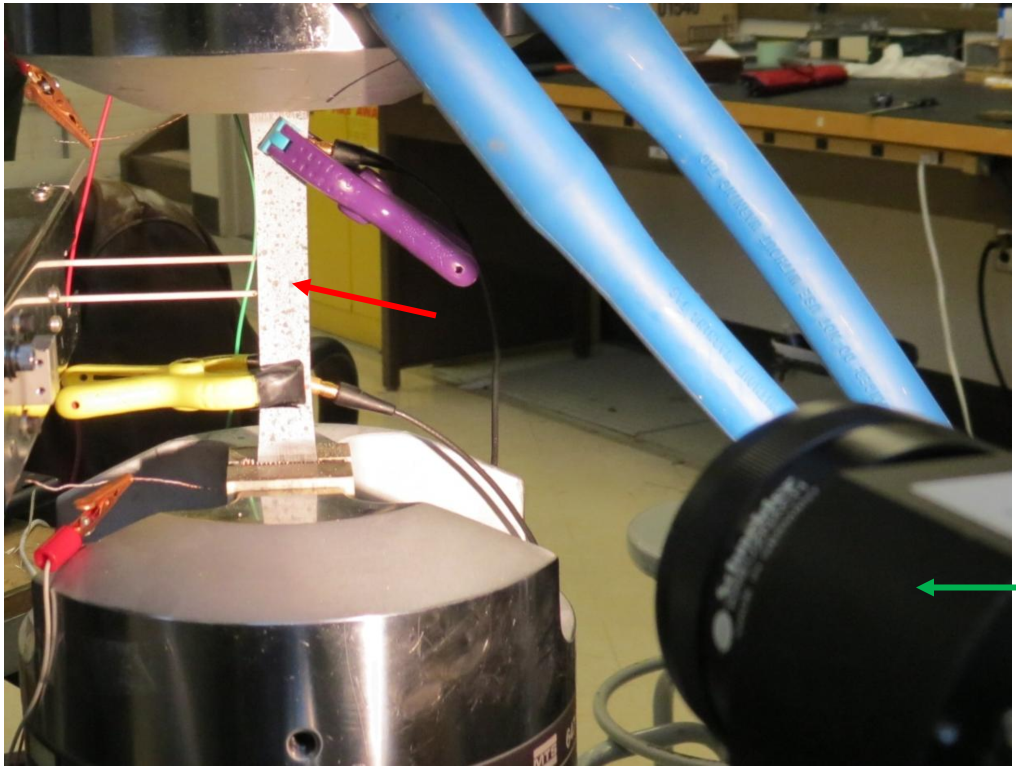


Acoustic Emission Sensors

Room Temperature Tensile Tests

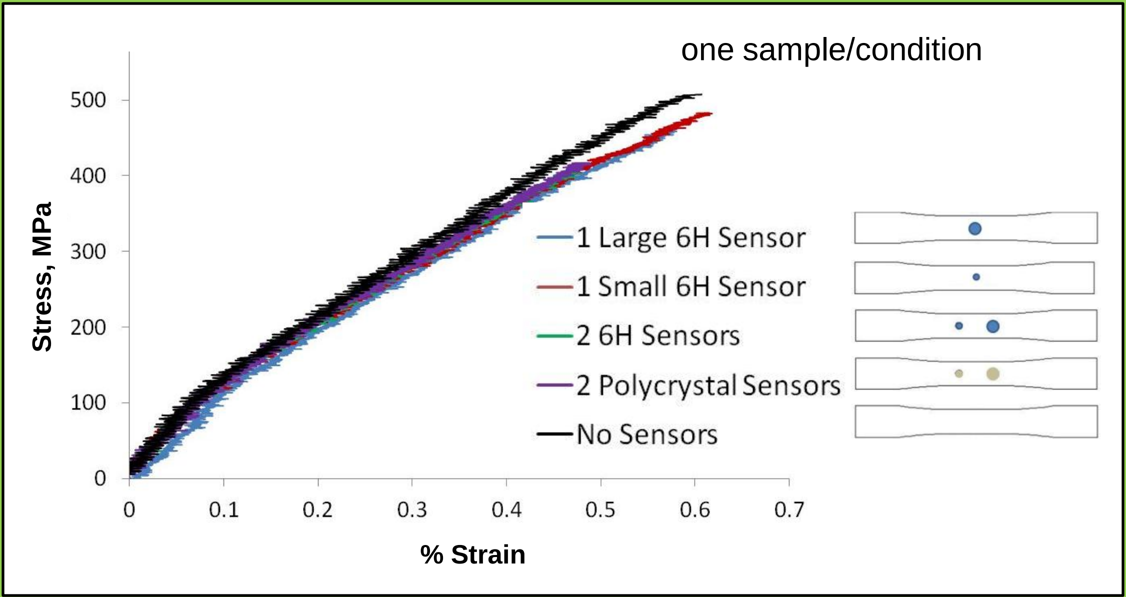
<u>Digital Image Correlation (DIC)</u>

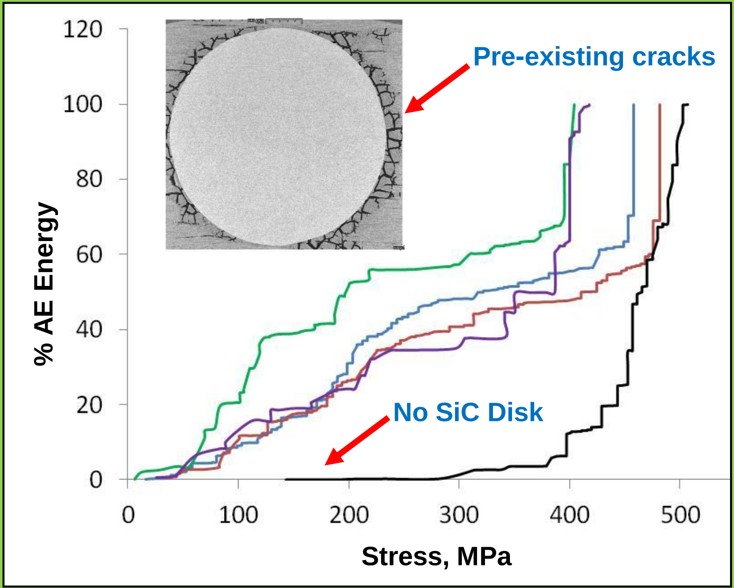
- A strain visualization method used to gain full-field strain information.
- A pattern is applied to the sample and digital cameras are used to track the displacement of the "points."
- This gives insight into crack development/progression that can't be achieved through use of conventional extensometers.



Digital camera: strain visualization

Room Temperature Tensile Test Results



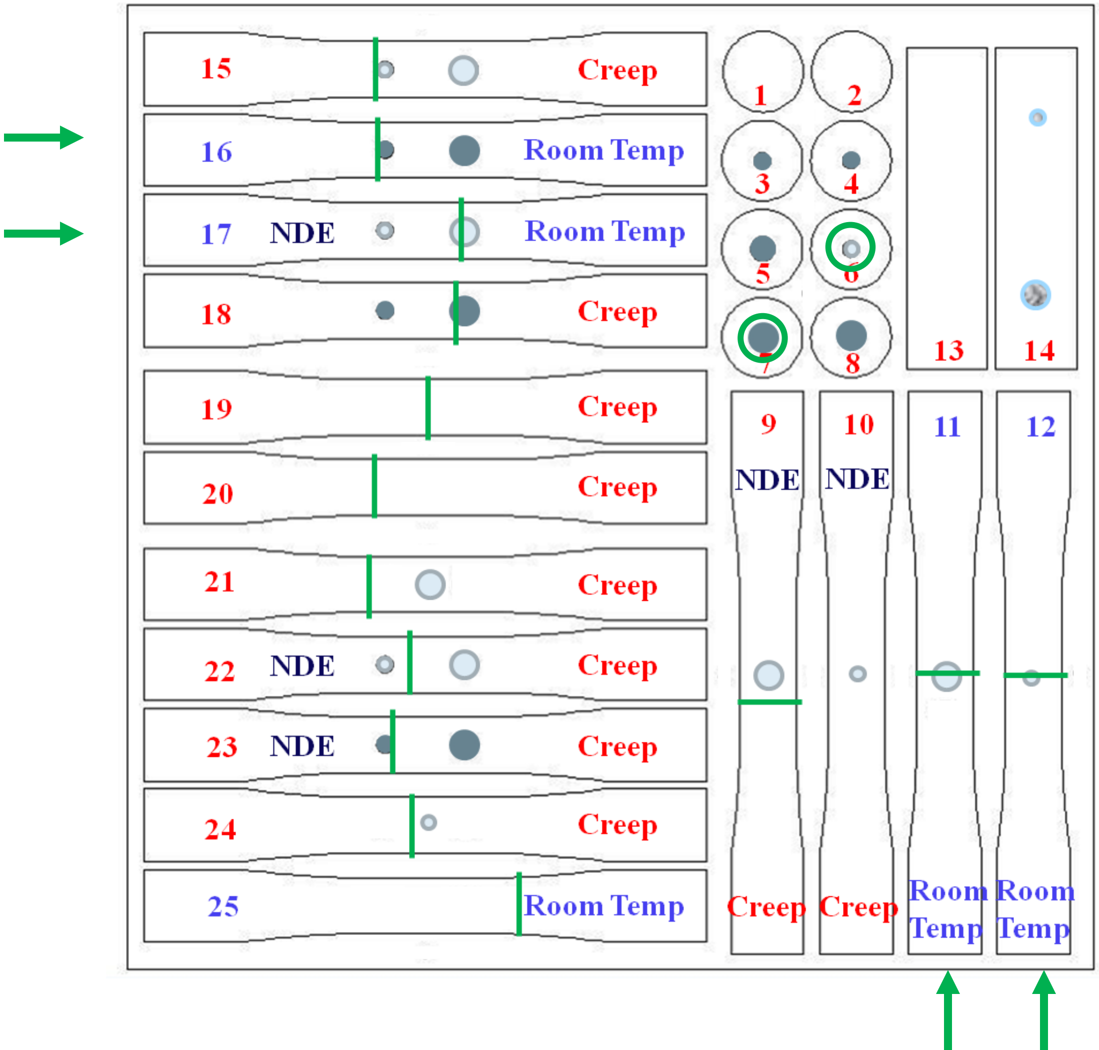


SIC Disks	E, GPa	0.005% Offset Stress, MPa	UTS, MPa	Ultimate Strain, %	First AE Stress, MPa	First Loud AE Stress, MPa
1 Large 6H	125	120	460	0.579	16	39.2
1 Small 6H	130	140	483	0.615	34	47.3
2 6H	130	135	406	0.476	6	10.6
2 Polycrystalline	125	140	417	0.486	26	47.7
NONE	135	150	508	0.607	143	313.7

- **Modulus was only slightly reduced for samples with SiC disks**
- **2-Disk samples had a 20% reduction in strength and strain**
- **Failure strain was similar for baseline and single SiC disk samples**
- **Baseline sample had significantly higher 1st AE stress**
- **All samples broke near the center or edge of a SiC disk**

Room Temperature Tensile Test Results: Fracture Behavior

- Green lines indicate the location of the fracture
- All room temperature samples w/SiC disks broke near a dummy sensor location



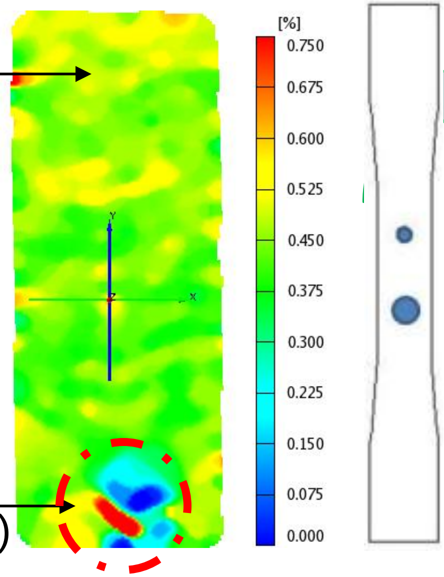
Room Temperature Tensile Test Results

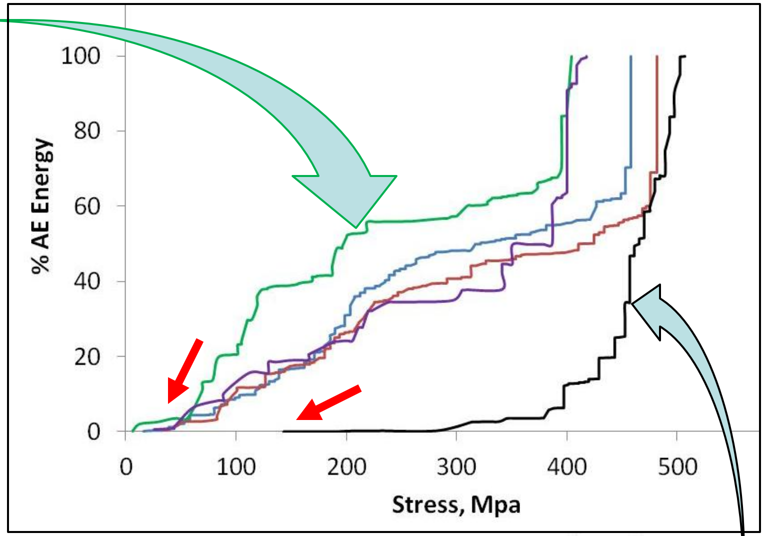
Digital Image Correlation (DIC) Indicated That Cracking Initiated Near SiC Disks

Small SiC Disk
(5 mm diameter)

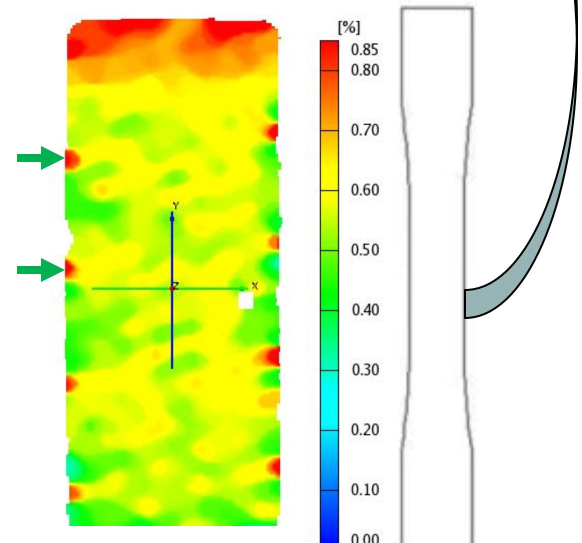
Sample 17

Large SiC Disk
(7.5 mm diameter)



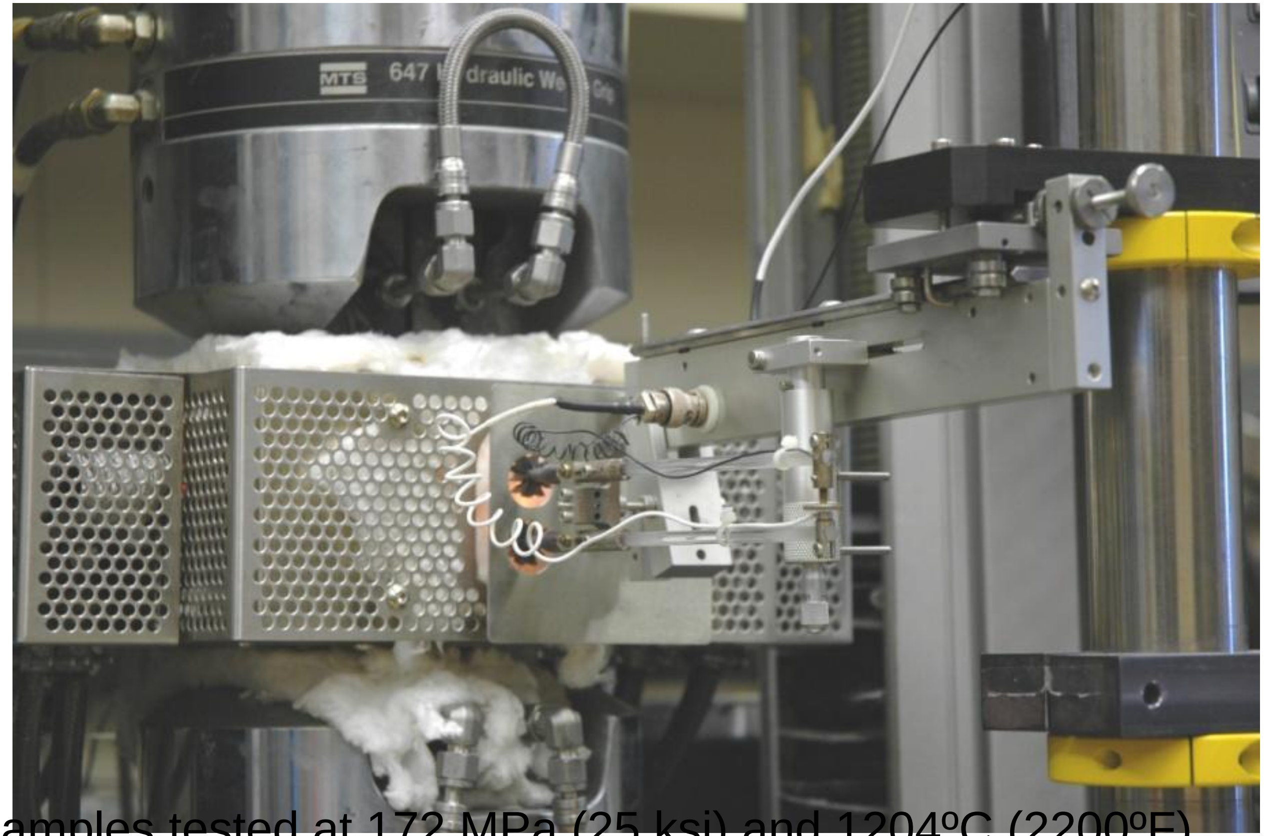


- Embedded SiC disk sample showed high local strain near the large disk, which is where failure occurred
- AE events initiated at much lower stress when SiC disks were present
- Baseline sample with no embedded SiC disks had a distribution of strain, with cracks forming at the edges



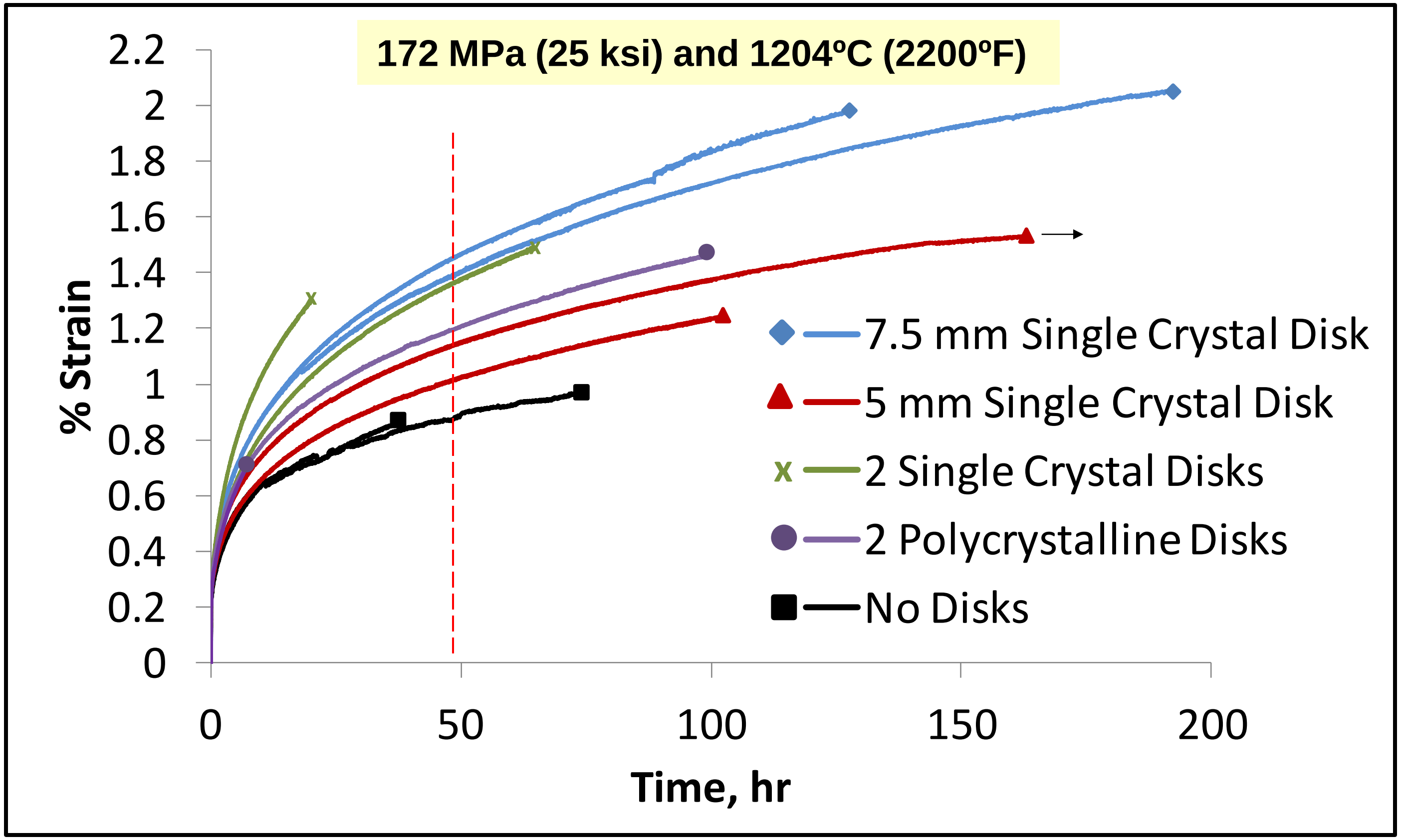
Sample 25

Tensile Creep Testing in Air



- All samples tested at 172 MPa (25 ksi) and 1204ºC (2200ºF)
- Strain measured with a capacitance probe extensometer (25.4 mm gage)
- Heated to 1204ºC and held
- Loaded to 172 MPa at a rate of 0.127 mm/min
- Load held constant until failure

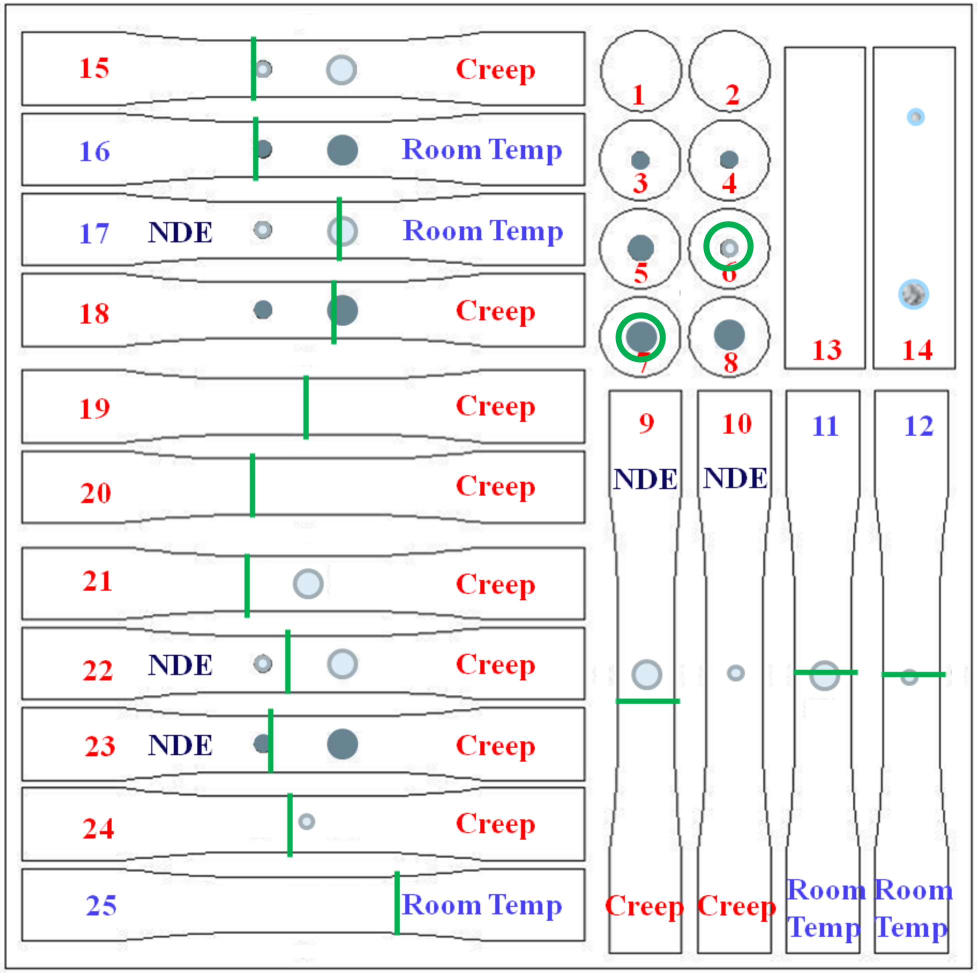
Tensile Creep Test Results



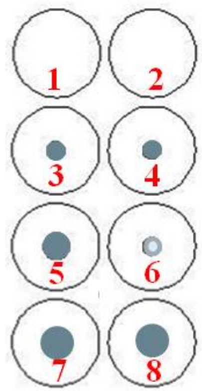
- **Samples with embedded SiC disks exhibited faster creep rates**
- **Compared to the baseline (no SiC disks), the time to failure was similar or longer for samples with SiC disks**
- **Sample lives > 50 hrs were achieved in most cases, but the impact of the total strain experienced by the composite needs assessed (wrt considering the lifetime of an embedded sensor).**

Tensile Creep Test Results: Fracture Behavior

- Green lines indicate the location of the fracture
- Most creep samples w/SiC disks broke near a dummy sensor location



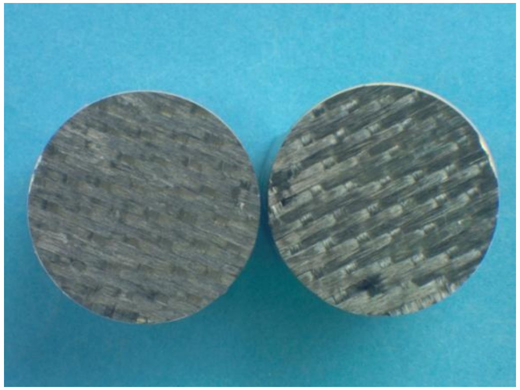
Room Temperature Interlaminar Tensile Tests



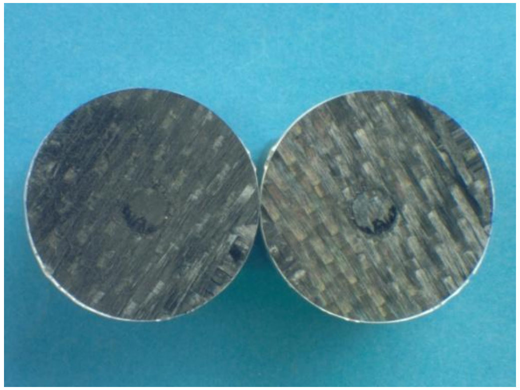
Type of ILS Sample	Strength, MPa	Fracture Location
No SiC Disk	24.2	In Composite
No SiC Disk	~~9.6~~	~~In Glue~~
Small Polycrystalline	26.3	In Composite
Small Polycrystalline	25.7	In Composite
Small 6H	21.7	At SiC Disk
Large Polycrystalline	18.0	At SiC Disk

- Small polycrystalline SiC disks had no effect on ILT strength (good bond)
- Small 6H single crystal SiC disks broke at the disk and had ~10% reduction in strength
- Large polycrystalline SiC disks broke at the disk and had ~25% reduction in strength

Baseline



Small 6H SiC Disk



Large Poly SiC Disk

Acknowledgments

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- D. Roth, NASA Glenn Research Center,
- R. Rauser, University of Toledo, Cleveland, OH
- N. Wilmoth, ASRC Aerospace Corp., Cleveland, OH

Summary and Conclusions

- In future work, changes will need to be made to prevent SiC disk/sensor cracking. Understanding the cause of the cracking is being pursued.
- X-radiography was useful for pinpointing the location of the dummy sensors, and computed tomography (CT) was a valuable tool for nondestructively evaluating the characteristics of the SiC disks and matrix in the CMC.
- Individual SiC disks caused a small reduction in room temp. tensile strength.
- Samples with two SiC disks had ≈20% room temperature strength reduction.
- Digital image correlation (DIC) and acoustic emission (AE) were used to evaluate the effects of the SiC disks on crack formation.
- The creep rate increased with the presence of SiC disks (for the test conditions evaluated). Note that large amounts of scatter are common in the creep testing of CMCs. In future efforts, more repeat tests will be performed.
- Microstructural evaluation to assess the interaction between the SiC disks and the composite matrix, including the stability of the metal coatings (W-Ni) on several of the disks is in progress.
- Given the difficulty of developing this type of embedded sensor technology, it is important to pursue the research well before the technology is needed.